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Inhibitive potential of benign Caesarweed leaves extract (CWLE) as corrosion inhibitor of aluminium in H₂SO₄ phase

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ABSTRACT

Caesar-weed leaves extract (CWLE) was employed as inhibitor on the corrosion of aluminium in 0.5M H_2SO_4 . The process was achieved by means of weight loss technique. The results revealed that inhibition efficiency increased with increase in concentration of the inhibitor but decreased with increase in temperature. Apparent activation energy increased with inhibitor concentration indicating the effectiveness of CWLE to retard the corrosion process by increasing the energy barrier. The corrosion process followed first order kinetics with correlation coefficients (R2) > 0.94. The data obtained were fitted into various adsorption isotherms, though Langmuir isotherm was found to be the best fit. The thermodynamic parameters revealed that the extract adsorption on aluminium surface was spontaneous, exothermic, and supported physical adsorption process.

Key words: Caesar-weed, corrosion, inhibition, aluminium, adsorption

1. INTRODUCTION

Corrosion of metals and metallic alloys in fragile acidic, alkaline and/or saline environment results in mechanical failure of equipment when exposed to these environments. Because of this, there is need for addition of corrosion inhibitors in minute quantities, to retard the corrosion process [1]. Corrosion inhibitors protect the metal surface through molecular interaction leading to physical or chemical adsorption [2].

Organic compounds containing heteroatoms such as N, S or O are effective corrosion inhibitors for many metals and alloys [3]. Addition of organic molecules with heteroatoms to corrosive environment helps in mitigating corrosion [4]. These molecules usually form



complexes by adsorbing to the metal surfaces [5-6].

Plant extracts are known to be rich in heterocyclic like alkaloids, constituents flavonoids, sapponins, tannins, phenols, steroids, etc. These constituents enhance their corrosion inhibitive properties [7]. Researchers have reported the use of plants extracts as corrosion inhibitors in different aggressive environments. Onion peel/skin [8-9], water hyacinth [10], Newbouldia laevis [11], Spondias mombin [12], Costus afer leaves and stem [13-14], Cucumber [15].

Corrosion resistance of aluminium and its alloy arises from its ability to form an oxide film with oxygen in the air. However, these oxide films readily undergo corrosion in harsh environment. Hence, the need to prevent its corrosion using green inhibitors. This study will therefore focus on the use of benign Caesarweed leaves extract in the inhibition of corrosion on aluminium in 0.5 M H_2SO_4 phase.

2. METHOD AND MATERIALS

2.1. Preparation of Caesar-weed leaves

Fresh samples of Caesar-weed leaves were collected from farmland in Aluu Ikwerre L.G.A. of Rivers State Nigeria. The sample were identified in Plant Science and Biotechnology Department, University of Port Harcourt. The samples were washed, air-dried at room temperature, and pulverized. A 100 g of sample was soaked in 500 ml of methanol for 7 days. The samples were filtered off from the extracted solution which was concentrated by distilling off the methanol. Inhibitor solutions were prepared by weighing the correct mass of the inhibitor (0.2 g, 0.4 g, 0.6 g, 0.8 g and 1.0 g) and dissolved in 100 ml of $0.5 \text{ M} \text{ H}_2 \text{SO}_4$ solution and make up to 1000 ml to obtain 0.2 g/L, 0.4 g/L, 0.6 g/L, 0.8 g/L and 1.0 g/L inhibitor solutions. The blank solution to be used will be the $0.5 \text{ M} \text{ H}_2 \text{SO}_4$ solution without the inhibitor added.

2.2. Specimen Preparation

Aluminium sheet used for this study was obtained from Science and Engineering Workshop of University of Port Harcourt, Rivers State of Nigeria. It was cut into rectangular coupons of dimensions 2 cm x 3 cm with thickness of 1.5 mm. The coupons, drilled at the centre for passage of thread, were mechanically polished to obtain a clean shiny surface, washed in deionized water, degreased in ethanol, dried in acetone, and then stored in moisture free desiccator for subsequent use.

2.3. Phytochemical Analysis

Phytochemical analysis of methanolic CWLE was carried out using standard procedures according to the method reported elsewhere [16].

2.4. Test for flavonoids

Crude extract was mixed with few fragments of magnesium ribbon and concentrated HCl was added drop wise. Pink or scarlet colour appeared after few minutes which indicated the presence of flavonoids

2.5. Test for saponins

Crude extract was mixed with 5ml of distilled water in a test tube and it was shaken vigorously. The formation of stable foam was



taken as an indication for the presence of saponins.

2.6. Test for steroid

Crude extract was mixed with 2ml of chloroform and concentrated H₂SO₄ was added sidewise. A red colour produced in the lower chloroform layer indicated the presence of steroids. Another test was performed by mixing crude extract with 2ml of chloroform. Then 2 ml of each of concentrated H₂SO₄ and acetic acid were poured into the mixture. The development of a greenish coloration indicated the presence of steroids.

2.7. Test for alkaloids

Crude extract was mixed with 2 ml of 1% HCl and heated gently. Mayer's And Wagner's reagents were then added to the mixture. Turbidity of the resulting precipitate was taken as evidence for the presence of alkaloids.

2.8. Test for reducing sugar

Fehling's test

Equal volume of Fehling A and Fehling B reagents were mixed together and 2 ml of it was added to crude extract and gently boiled. A brick red precipitate appeared at the bottom of the test tube indicated the presence of reducing sugars.

2.9. Test for phenols and tannins

Crude extract was mixed with 2ml of 2% solution of FeCl₃. A blue-green or black coloration indicated the presence of phenols and tannins

2.10. Gravimetric Analysis

In the weight loss experiment, the pre-cleaned aluminium coupons were completely immersed in 100 ml of the respective inhibitor/blank solutions maintained at 303 K, 313 K, 323 K, and 333 K in a thermo-stated water bath. The weight loss was determined by retrieving the coupons at 24 hrs intervals progressively for 168 hrs. The retrieved samples were washed under running water with bristle brush, rinsed in ethanol and dried in acetone solution. The samples were then reweighed to determine the weight loss of each sample.

The weight loss was calculated in grams as the difference between the initial weight prior to immersion, and weight after the removal from acid solution. Triplicate experiments were performed in each case and the mean values reported to the nearest 0.0001g on an AB54 AR analytical weighing balance.

The inhibition efficiency in percentage (I. E.%) was computed using equation 1:

%Inhibition Efficiency =
$$\frac{W_b - w_i}{w_i} \ge 100\%$$
 ...eq 1

Where:

 W_b and W_i are the weight loss of aluminium metal coupons without and with inhibitor respectively.

The corrosion rate (W) was computed using equation 2

Corrosion rate (g/cm2/day)
$$CR = \frac{\Delta W}{At}$$
 ...eq. 2

Where:

A is Area (cm2) of coupon exposed, t is immersion time in hours ΔW is the change in weight or weight loss (g).



 ΔW is the difference between initial weight of coupon before immersion and the final weight of coupon after immersion.

The degree of surface coverage was computed using equation 3

Surface coverage=
$$\frac{I E \%}{100}$$
 ...eq. 3

3. RESULT AND DISCUSSION

3.1. Phytochemical Analysis

Phytochemical analysis of Caesar-weed leaves extract CWLE showed presence of some phytochemicals shown in Table 1 which may be responsible for its inhibition efficiency.

3.2. Weight loss study

The higher the concentration of CWLE inhibitor, the lower the weight loss as can be seen from the graph. This is as a result of adsorption of the phytochemicals from the CWLE inhibitor onto aluminium surface which covers the surface thereby preventing direct contact between aluminium and the corrodent solution. This therefore retards the corrosion process.

The results strongly revealed that the corrosion of aluminium decreased significantly in presence of CWLE. This arose due to the fact that addition of the inhibitor extract provided electrons for the reduction of H+ to hydrogen gas through the double bond or heteroatoms. The inhibitor reduced corrosion by adsorbing on the metal surface thereby protecting the surface by forming a barrier film on the surface [17] [18]. The adsorption of such compounds on the metal surface creates a barrier for charge and mass transfer thereby minimizing corrosion reaction [19].

3.3. Inhibition Efficiency

Generally, increase in temperature increases the rate of corrosion reaction and reduces the inhibition efficiency of an inhibitor. In addition, increasing the concentration of an CWLE inhibitor, means increasing the amount of inhibitor which will adsorb on the aluminium surface thereby retarding corrosion and increasing inhibition efficiency. More the adsorbed inhibitors on the metal surface, more they will form protective barrier films which prevent direct contact between the acid and the metal, thereby increasing inhibition efficiency. The result revealed that the inhibition efficiency of CWLE decreased significantly as the temperature increased as shown in Figure 5 [20-21]. However, the corrosion inhibition efficiency increased with increase the CWLE in concentration indicating the presence of

Table 1. Phytochemical composition of methanolic extract of Caesar-weed						
Qualitative availability						
+						
+						
+						
-						
+						
-						
-						





Figure 1. (A) Variation of weight loss with time of Al in 0.5 M in H_2SO_4 at 303K in the presence of different concentration of CWLE.

Figure 2. (B) Variation of weight loss with time of Al in 0.5 M in H_2SO_4 at 313K in the presence of different concentration CWLE.





Figure 4. (D) Variation of weight loss with time of Al in 0.5 M in H_2SO_4 at 333K in the presence of different concentration of CWLE.

phytochemicals in the extract [22-23].

3.4. Corrosion rate

Effectiveness of CWLE in reducing corrosion of aluminium in 0.5 M H_2SO_4 was obtained by comparing the corrosion rate of aluminium in the presence and absence of CWLE inhibitor.

As can be seen from Figure 6, CWLE retards the corrosion rate by inhibition of the process.

The lowest values for corrosion rate were observed for 1.0g/L. while the highest value was for 0.2 g/L. The corrosion reaction was inhibited (Figure 6) showed the variation of Corrosion rate with inhibitor concentration at 303K – 333K. Corrosion rate increased with temperature rise but decreased with increase in inhibitor concentration. This is in conformity with earlier results reported by [24-25].

3.5. Kinetic study





Figure 5. (E) Variation of inhibition efficiency of CWLE for corrosion of Al in 0.5 M in H_2SO_4 with different inhibitors concentration at different temperature.

Figure 6. (F) The relationship between corrosion rate and concentrations of CWLE at different temperature.



Figure 7. (G) Variation of – log (Weight Loss) with time for corrosion of Al in 0.5M H₂SO₄ containing various concentrations of CWLE of at 303K.

Figure 8. (H) Variation of $-\log$ (Weight Loss) with time for corrosion of Al in 0.5M H₂SO₄ containing various concentrations of CWLE of at 313K.

The kinetics of CWLE as an inhibitor in H_2SO_4 solution was investigated by fitting the obtained data from weight loss measurements into equation 3 [26].

-log (weight loss) =
$$\frac{k_1 t}{2.303}$$
 ...eq. 4

Where:

 k_1 is the first order rate constant and t is time in days.

The plots of variation of $-\log (weight loss)$ against time for aluminium corrosion in 0.5 M H₂SO₄ solution in the presence of various concentrations of CWLE at 303K - 333K were shown in Figures 7-10.

The linear plots with R_2 values close to unity show the fitness of the data to first order kinetics. The half-life of first order reaction is related to the rate constant as follows:





Figure 9. (I) Variation of – log (Weight Loss) with time for corrosion of Al in 0.5M H₂SO₄ containing various concentrations of CWLE of at 323K.

Figure 10. (J) Variation of –log (Weight Loss) with time for corrosion of Al in 0.5M H₂SO₄ containing various concentrations of CWLE at 333K.



Figure 11. (K) Arrhenius plots in the presence and absence of different concentrations of CWLE on Al inhibition.

Figure 12. (L) Eyring Transition state plots in the presence and absence of different concentrations of CWLE on Al inhibition.

$$t_{1/2} = \frac{0.693}{k_1}$$
 ...eq. 5

Values of k_1 , $t_{1/2}$ and R_2 obtained from the kinetic plots are recorded in Table 2.

The $t_{1/2}$ analysis revealed that for each concentration, the half-life of aluminium in 0.5 M H_2SO_4 corrodent solution decreased with increase in temperature and that R_2 tends to unity, hence CWLE was a good inhibitor for

aluminium in 0.5 M H_2SO_4 solution [27]. The optimum half-life was achieved at 303 K.

3.6. Effect of temperature

The Eyring transition state equation (6) and Arrhenius equation (7) were used in calculating activation parameters such as activation energy E_a , activation enthalpy change ΔH^* and activation entropy change ΔS^* for the corrosion process.



Table 2. Kinetic parameters for corrosion of Al in $0.5M H_2SO_4$ containing various concentrations of CWLE at 303K - 333K.

Concentrat ion of CWSE (g/L)	(days ⁻¹)			t _{1/2} (days)			R ²					
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
Blank	0.1149	0.171	0.202	0.21	6.03	4.06	3.44	3.1	0.993	0.949	0.992	0.957
0.2	0.1011	0.18	0.219	0.221	6.85	3.85	3.16	3.14	0.98	0.95	0.995	0.959
0.4	0.1102	0.189	0.216	0.22	6.29	3.67	3.2	3.15	0.976	0.957	0.994	0.963
0.6	0.1047	0.184	0.217	0.22	6.62	3.76	3.19	3.15	0.978	0.954	0.993	0.959
0.8	0.0992	0.191	0.228	0.232	6.99	3.63	3.04	2.98	0.944	0.962	0.997	0.955
1	0.1385	0.197	0.236	0.232	5	3.52	2.93	2.98	0.99	0.954	0.99	0.968

Table 3. Corrosion activation parameters for Al in 0.5M H₂SO₄ in the absence and presence of different concentrations of CWLE inhibitor.

E _a (kJ/mol)	ΔH* (kJ/mol)	ΔS* (J/mol/K)
45.95	43.30	-74.34
54.45	51.78	-54.02
58.17	55.50	-43.27
61.78	59.11	-32.65
61.96	59.30	-33.58
65.48	62.80	-24.36
	Ea (kJ/mol) 45.95 54.45 58.17 61.78 61.96 65.48	E _a (kJ/mol)ΔH* (kJ/mol)45.9543.3054.4551.7858.1755.5061.7859.1161.9659.3065.4862.80

$$\log \frac{\mathrm{CR}}{\mathrm{T}} = -\frac{\Delta \mathrm{H}^*}{2.303\mathrm{RT}} + \left(\frac{\Delta \mathrm{S}^*}{2.303\mathrm{R}} + \log \frac{\mathrm{R}}{\mathrm{Nh}}\right) \quad eq. \ 6$$

$$\log CR = \log A + \left(-\frac{Ea}{2.303RT}\right) \qquad eq. 7$$

Where:

T is the temperature in kelvin,

R is the universal gas constant (8.314 J/mol/K), N is the Avogadro's number ($6.02 \times 1023 \text{ mol-1}$), and h is the Planck's constant (6.626×10^{-34} Js).

A plot of log $\frac{CR}{T}$ against $\frac{1}{T}$ gives a straight line with intercept $(\frac{\Delta S^*}{2.303R} + \log \frac{R}{Nh})$ and slope $(-\frac{\Delta H^*}{2.303R})$ while for Arrhenius a plot of log CR against $\frac{1}{T}$ gives a straight line with intercept (log A) and slope $(-\frac{Ea}{2.303R})$ The activation thermodynamic evaluations revealed that the values of Ea increased with increase in the inhibitor's concentrations but greater in the presence of CWLE inhibitor than in blank. This increment in E_a with inhibitor showed physical adsorption [28] and revealed that the corrosion process was inhibited by CWLE. The negative values of activation entropies (ΔS^*) reveals non-spontaneity of the activation complex. Entropy of activation in the presence of the inhibitor increases than in its absence. Thus, implying this there is increase in disorderliness in going from reactant to activated complex [29]. The enthalpies of activation were all positive indicating the endothermic nature of aluminium dissolution. Higher enthalpies of activation (ΔH^*) in the





presence of the inhibitors than in its absence, makes aluminium dissolution difficult [30-31]. This could be attributed to higher energy barrier because of higher efficiency of protection offered by the adsorbed inhibitor [32].

3.7. Adsorption Isotherm/Thermodynamics

Adsorption of plant extracts on the metal surface prevents further corrosion of the metal by blocking the active sites on the surface of the metal [33].

The degree of surface coverage values for CWLE on the aluminium surface was obtained using equation 3. The results were fitted into adsorption isotherm equations such as Frumkin, Temkin, Henry, Freundlich, Langmuir, El-Awady, Virial-Parson and Flory-Huggins. The correlation coefficient (R2) was used to determine the best fitted isotherm (Table 4). The values of R₂ obtained in the Langmuir isotherm are closest to unity indicating that the adsorption of CWLE on to aluminium surface best fits the Langmuir isotherm. From the equilibrium adsorption constant K_{ads}, Gibb Free energy of adsorption ΔG°_{ads} , was calculated using the equation 12.

 $\Delta G^{\circ}_{ads} = -2.303 \text{ RTlog } K_{ads} \qquad ..eq. 8$

The enthalpy and entropy of adsorption of CWLE on aluminium surface can better be explained using the Gibb's free energy equation:

$$\Delta G^{\circ}ads = \Delta H^{\circ}ads - T\Delta S^{\circ}ads \qquad ..eq. 9$$

To calculate the enthalpy of adsorption ΔH°_{ads} and entropy of adsorption ΔS°_{ads} , ΔG°_{ads} (of best fitted isotherm -Langmuir isotherm) was plotted against T (Figure 14) and a straight line was obtained with intercept equal ΔH°_{ads} and slope equal to ΔS°_{ads} . The calculated values of enthalpy and entropy of adsorption were displayed in Table 5

The negative value of ΔH°_{ads} reveals that the adsorption of CWLE on to aluminium surface was exothermic. Since the value of ΔH°_{ads} was less than 40 kJ/mol, the adsorption mechanism followed a physisorption process [34]. The entropy of adsorption value is positive, suggesting that the adsorption was accompanied by an increase in entropy ΔS°_{ads} . The values of ΔG°_{ads} were all negative indicating spontaneous and physical adsorption process.

4. CONCLUSION



Table 4. Adsorption parameters from different isotherms at 303 K-333 K.							
Isotherm	Temperature (K)	R ²	Slope	Intercept	Α	Kads	ΔG° _{ads} (kJ/mol)
Frumkin	303	0.9845	5.33111	-3.67211	2.66556	0.0254	-0.865
	313	0.9664	5.55934	-3.67425	2.77967	0.0254	-0.865
	323	0.9404	4.53376	-2.80333	2.26688	0.0606	-3.258
	333	0.9083	5.40498	-3.09653	2.70249	0.0452	-2.547
Temkin	303	0.9694	0.30644	0.79872	-3.7577	404.02	-25.241
	313	0.9124	0.25583	0.73705	-4.5010	760.33	-27.720
	323	0.8386	0.31311	0.68201	-3.6776	150.73	-24.254
	333	0.8149	0.22421	0.59406	-5.1358	446.27	-28.015
					f		
Virial- Parson	303	0.9582	-0.37668	0.76206	3.0570	105.46	-21.857
	313	0.8791	-0.30175	0.69587	3.8161	202.35	-24.274
	323	0.7451	-0.38067	0.61321	3.0249	40.82	-20.750
	333	0.7369	-0.25976	0.53189	4.4329	111.58	-24.177
					x		
Flory-	303	0.8926	1.57331	1.0088	1.57331	10.2047	-15.972
Huggins	313	0.8292	2.10547	1.12122	2.10547	13.2197	-17.173
	323	0.6934	1.66014	0.71779	1.66014	5.2214	-15.227
	333	0.6893	2.81896	0.94208	2.81896	8.7514	-17.128
					1/y		
El-Awady	303	0.9371	0.65511	0.59202	1.5265	3.9086	-13.554
	313	0.8915	0.50432	0.44652	1.9829	2.7959	-13.130
	323	0.8246	0.57626	0.33311	1.7353	2.1533	-12.848
	333	0.8087	0.39548	0.16633	2.5286	1.4667	-12.182
Langmuir	303	0.9991	0.80845	0.09589		0.8019	-9.563
	313	0.9970	0.83096	0.13170		0.7384	-9.664
	323	0.9866	0.77009	0.16589		0.6825	-9.762
	333	0.9912	0.81713	0.22629		0.5939	-9.796
Freundlich	303	0.9839	0.19158	-0.09590		0.8019	-9.563
	313	0.9323	0.16901	-0.13175		0.7383	-9.664
	323	0.8680	0.22997	-0.16587		0.6825	-9.762
	333	0.8495	0.18287	-0.22629		0.5939	-9.796
Henry	303	0.9897	0.27025	0.54981		0.27025	-6.823
	313	0.9824	0.23170	0.52560		0.23170	-6.648
	323	0.9567	0.29190	0.41822		0.29190	-7.480
	333	0.9465	0.21090	0.40404		0.21090	-6.812

Caeser-weed leaves extract (CWLE) acted as a good inhibitor for corrosion of aluminium in 0.5 M H_2SO_4 . Inhibition efficiency increased with increase in concentration of the inhibitor but decreased with temperature. Apparently, the

activation energy increase with inhibitor concentration indicating the effectiveness of CWLE to retard the corrosion process by increasing the energy barrier. The adsorption of the inhibitor best fitted the Langmuir isotherm



Table 5. Enthalpy and Entropy of adsorption values of CWLE on aluminium from ΔG° ads plot against temperature.

Slope	intercept	ΔH° _{ads} (kJ/mol)	ΔS°_{ads} (J/mol/K)
-0.00797	-7.16	-7.16	7.97

model equation. Values of entropy change, enthalpy change, and Gibbs free energy change of the adsorption are positive, negative, and negative respectively indicating spontaneous, endothermic, and feasible process accompanied by increase in disorderliness.

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6. CONFLICT OF INTEREST

The authors have declared that there is no conflict of interest.

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NA

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